National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1641d

Mercury in Water
(Acidified to 2% Nitric Acid)

This Standard Reference Material (SRM) is intended for the calibration of instruments and techniques used for the determination of mercury in natural waters. It is designed for the preparation of calibration solutions and for use as a "spike" sample in a "method-of-additions" analytical procedure. A unit of SRM 1641d consists of 10 ampoules, each ampoule containing approximately 10 mL of solution comprised of a trace amount of mercury in 2 % (v/v) nitric acid, initially stabilized with 1 mg/kg gold.

The mercury content in this SRM was certified using flow injection cold vapor atomic absorption spectroscopy (CVAAS) and isotope dilution inductively-coupled plasma mass spectrometry (ID-ICPMS). The certified mercury content and its estimated uncertainty are:

Table 1. Certified Value of Mercury (mass fraction)

1.590 mg/kg ± 0.018 mg/kg

The uncertainty in the certified value is ku_c, where k is the coverage factor for a 95 % confidence level and u_c is the "combined standard uncertainty" calculated according to the ISO Guide [1] and the procedure of Schiller and Eberhardt for combining independent analytical methods [2].

Notice to Users: At or below the mg/kg level, mercury solutions are not stabilized adequately with mineral acid alone. The addition of trace quantities of gold (~1 mg/kg) to the nitric acid solution of mercury provides greater stability. The gold may plate out on the ampoule wall during the period for which the certification is valid. Experience with the four previous issues of this SRM indicates that this has no effect on the mercury concentration.

Expiration of Certification: The certification of this SRM is valid within the measurement uncertainty specified, until **01 October 2009**, provided the SRM is handled in accordance with instructions given in this certificate.

Maintenance of Certification: The stability of this SRM will continue to be monitored over the period of certification and any substantive change in the certified value will be reported to customers. Return of the attached registration card will facilitate notification.

Technical coordination of SRM 1641d production was provided by J.D. Fassett of the NIST Analytical Chemistry Division. This SRM was prepared by B.R. Norman and certification analyses were performed by S.E. Long and S.J. Christopher of the NIST Analytical Chemistry Division.

The statistical evaluation of certification data was provided by H.K. Liu of the NIST Statistical Engineering Division.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 30 December 1999

Thomas E. Gills, Director
Office of Measurement Services

CAUTION: Traces of mercury vapor are present in most laboratory environments. Therefore, contamination of reagents, equipment, and common laboratory materials may cause a severe blank or background problem. Apparatus for analyses at and below the mg/kg level must be scrupulously cleaned immediately before use, and only the purest reagents with respect to mercury should be used.

Preparation of the SRM Solution: The polyethylene drum used to prepare SRM 1641d was first filled with a 10 % nitric acid solution containing approximately the same mercury and gold concentration as the SRM solution and conditioned with this solution for several weeks. The drum was then flushed with distilled water. SRM 1641d was prepared by filling the drum with approximately 200 L of distilled water, and acidifying to 2 % (v/v) nitric acid with high purity acid. Then spikes of high purity gold dissolved in aqua regia and high purity mercury dissolved in concentrated nitric acid were added sequentially, with thorough mixing. Finally, the bulk solution was ampouled. The density of the solution at 22 ^{o}C was 1.007 g/mL.

INSTRUCTIONS FOR USE

Ampoules are to be opened immediately before use by breaking the glass at the score line in the narrowest segment of the neck of the ampoule. Ampoules should not be resealed, nor stored in some other manner for subsequent use. Once ampoules are opened, dilutions should be prepared and used without delay since stability of the dilutions cannot be guaranteed. Blank determinations should be made of the diluent reagents. In the certification process at NIST, the samples were diluted by a factor of 1:400 in two steps using a 3 % nitric acid solution containing 0.05 % potassium dichromate as the diluent, in order to stabilize the mercury and to fall within the linear range of the instrumentation.

Preparation of Standard Solutions by Mass: Diluted working standard solutions can be prepared by transferring an aliquot of the SRM to an empty, dry, preweighed polyethylene bottle, and then reweighing the bottle. An appropriate dilute acid must be added by mass to bring the solution to the approximate desired dilution. The dilution need not be exact since the mass of the empty bottle, mass of the bottle plus SRM aliquot, and the final diluted mass of the solution will permit calculation of the exact concentration of the working solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true concentration in vacuum and will be in mg/kg units. Volumetric dilutions are **NOT** recommended due to uncertainties in volume calibrations and variations in density.

Preparation of Standard Solutions by Volume: Dilutions may be made by the addition of accurately measured aliquots, withdrawn from the just opened ampoule, to known volumes of an appropriate dilute acid using conventional techniques. The volumetric apparatus used should be scrupulously cleaned. The reliability of the dilution process will depend on the care exercised and on the reliability of the calibration of the volumetric apparatus used.

REFERENCES

[1] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994); (available at http://physics.nist.gov/Pubs/).

[2] Schiller, S.B. and Eberhardt, K.R., "Combining Data from Independent Analysis Methods," Spectrochimica, Acta, **46B** No. 12, pp. 1607-1613, (1991).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Telephone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.